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TWO REQUISITES OF A SOLVENT EXTRACTOR

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2—It must be simple enough to run so that technical experts are not required to operate it.

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Journal of Oil and Fat Industries

Devoted to Oils, Fats and Allied Industries

VOL. III JULY, 1926 No. 7

EDITORIAL

LET'S PROVE THAT TOM WAS RIGHT

Since its organization in 1912, our Society has enjoyed a healthy, vigorous growth. Its purposes and its accomplishments are of such a character as to enlist the interest and support of chemists all over the country, and that this interest and support really has been enlisted is perfectly obvious to anyone who is in close touch with the situation.

But still the enrollment falls far short of what it should be. Instead of some few hundreds, the members should number well above the thousand mark. There is any quantity of chemists in the United States employed in refineries, soap plants, etc., who are not A.O.C.S. members but who should be. They are interested in the work and would help themselves and others by affiliating with it.

It seems to be the general impression that the Chairman of the Membership Committee is the one to dig up new members. A little reflection would put an end to that idea—one individual could not possibly do the work alone. But even if he could, think of the greater results that could be accomplished if each and every chemist put his shoulder to the task! Each one of us knows some man or men who should be deeply interested in the work, and if we all put our proposition up to these men, the results would be immediate and astonishing.

One need only take the case of the tallow and grease chemists. These never have had any standards for use in reading their products, and now they are becoming interested in the Lovibond color system and glasses. Here is an opening for the Society.

The Journal certainly is a valuable paper now and it offers an entree into many laboratories. If we make the Journal a true expression of the

Society's activities, and if we *use* it, we will get into the Society men who otherwise might not join.

Tom Caldwell, one of the best secretaries the Society ever had, a fine worker, always had the idea that if the members believed in the Society thoroughly they would get new members soon enough. Let's prove that Tom was right!—H. P. T.

A WORTH WHILE PRIZE

At the spring meeting of the Society in New Orleans, it was proposed that a prize be awarded for the best manuscript submitted to the Journal. The suggestion met with instant and whole-hearted response. Without any appeal for funds being made, several members present subscribed a substantial sum of money as the basis of the fund.

The Committee has under consideration now defining rules, selecting judges, and completing the fund. It is hoped that complete and definite announcement can be made in the August number of the Journal.

The prize, or prizes—there may be more than one—will be substantial, and the contest most certainly should stimulate the discussion of highly important and interesting matters in which the Society is interested. Readers of the Journal should watch for the Committee's announcement—and in the meantime sharpen their pencils, neb their pens, clean typewriters, or set in order whatever may be their favorite instrument for transcribing.

OIL EXTRACTION IN THEORY AND PRACTICE

By LOUIS C. WHITON

The following paper was presented before the seventy-first meeting of the American Chemical Society. Because of its importance to the Oil and Fat Industries, permission has been obtained to print the paper concurrently with Industrial and Engineering Chemistry. Mr. Whiton makes this the introduction to a more detailed treatment of his topic, which will be published exclusively in the Journal of Oil & Fat Industries of July and August.—*The Editor.*

The United States has been singularly behind in the development of the use of solvent extraction for the manufacture of oil from oleaginous-bearing seeds. No doubt this has been due to the fact that in the early days, before a very extended use had been found for vegetable oils, there were considerably larger quantities of oil available in the seed than could be sold. Hydraulic pressing which recovered a relatively small proportion of the oil was therefore the logical line of development.

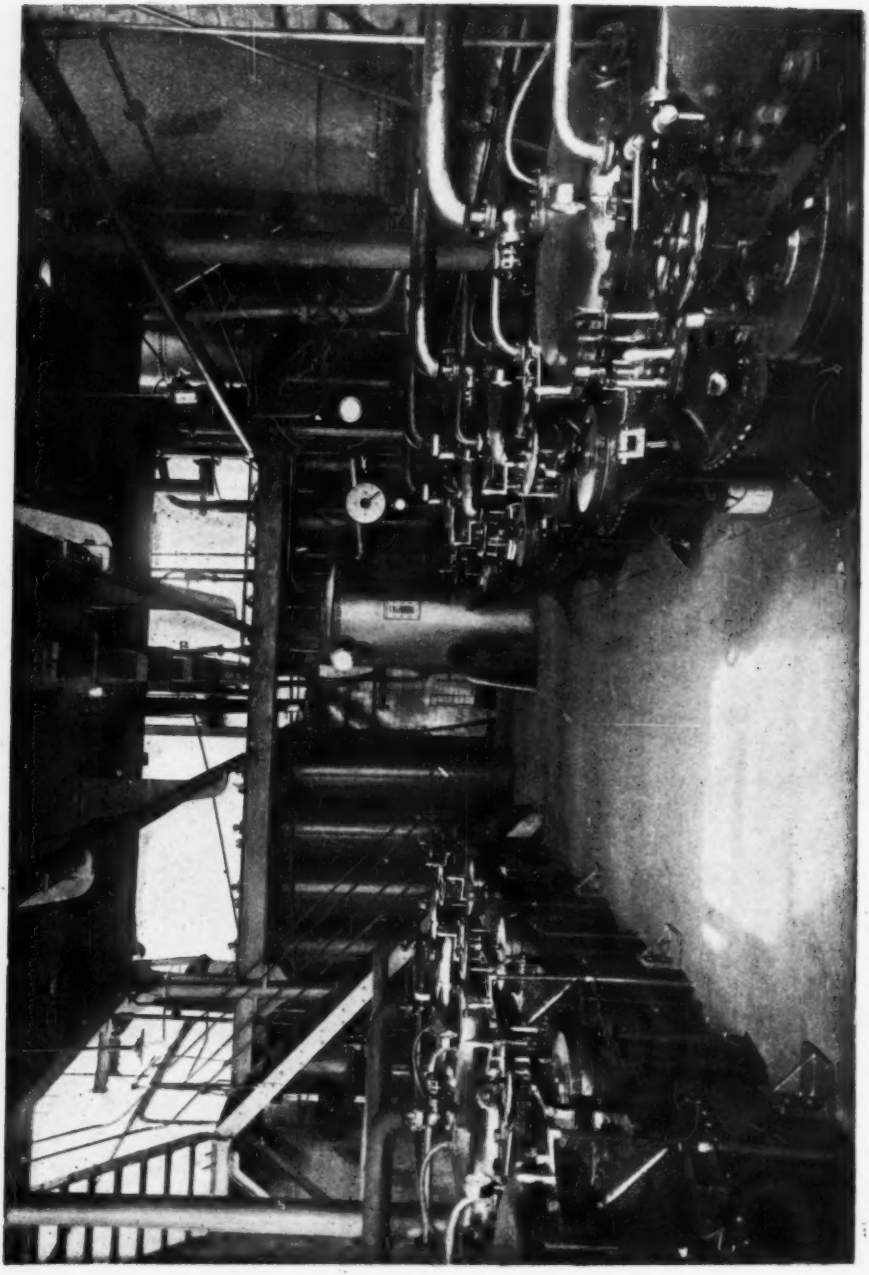
In Europe the same story could be told; but since 1900 the demand for vegetable oils has considerably increased and the conservation of this supply was forced upon the owners of the hydraulic press plants who had been wasting valuable product and selling it in the meal at about one-fourth of its real value as oil. Many of them turned to solvent extraction and it is now a highly developed industry abroad, particularly in the Mediterranean countries.

As is well known, there has been somewhat of an upheaval in the vegetable oil industry in the United States in the last five years. Changed labor conditions due to the war may have had something to do with it, but the fact remains that the stage in the development of this industry has been reached when it has become unprofitable and shortsighted to give away 100 to 150 lbs. of an oil worth about 10c. a pound, per ton of meal which is worth about 2½c. a pound. The discussion will be left to others as to the value of this fat as animal food—that is somewhat beside the question—the important point is that the oil can be used to a considerably greater advantage elsewhere and sold for four times more than if it is used as cattle feed.

In order that the solvent extraction plant be a success in replacing the hydraulic press, it is necessary for it to fulfill the following requirements:

1. It must produce absolutely solvent-free oil and meal so that the former may be used for the refinery and the latter as cattle food.
2. It must be as free from complications as possible so that expensive technical supervision is not required.
3. The machinery must be of such a nature that small plants of the size of the average cotton oil plant in the South can be run at a profit for a short season.

To anyone who has ever had the occasion to review the patents on solvent extraction, he has found it to be one of the favorite playthings of



Early form of Bataille Extractors Installed in England in 1892

the theorist; but contrary to many types of machinery, solvent extraction almost resolves itself to the crude formula: the fewer the refinements, the better the extractor. In other words, the laboratory Soxhlet on a commercial scale is almost the ideal. Below are mentioned some of the complications which seem reasonable to the best engineers until they have had experience in the solvent extraction field.

Freeing Oil of Solvent

It seems more reasonable to collect the oil bearing solvent from a number of extractors and to distill this from a large still. This appears to require less labor and a less costly installation. From a practical standpoint it heats the oil too long a time, thus producing an inferior quality of oil, and requires intensive steaming and heating at the end to eliminate traces of solvent from a large quantity of residual oil. It is far better to use small stills connected with each extractor and to have each still finish off the oil from a single extraction. The oil will thus be less violently treated and of a better quality. Since a still requires no labor to speak of, there is no disadvantage from that standpoint. The idea that oil cannot be freed of solvent is illogical when one realizes that products much more difficult to remove are eliminated in a refinery deodorizer and that the oil still operates upon the same principle; that is, it must separate an oil, boiling at a high temperature, from a low boiling solvent which, needless to say, should be properly rectified beforehand.

Freeing Meal of Solvent

The elimination of the solvent from the meal is also accomplished by steaming. A natural conclusion is that this solvent can be more easily volatilized if the material is stirred. This is partially true. Nevertheless consider the mechanical difficulties of stirring several tons of solid particles of meal which is not surrounded by liquid. It requires a fair amount of power, offers possible leaks at stuffing boxes and is a movable part which it is wise to dispense with if possible. The only successful method of avoiding the agitator is one used by one of the manufacturers of extractors and which consists in draining the meal thoroughly of solvent and then introducing solvent vapors so that there will be a large quantity of vaporous material to steam rather than liquid which first must be vaporized.

Discharging the Extractor

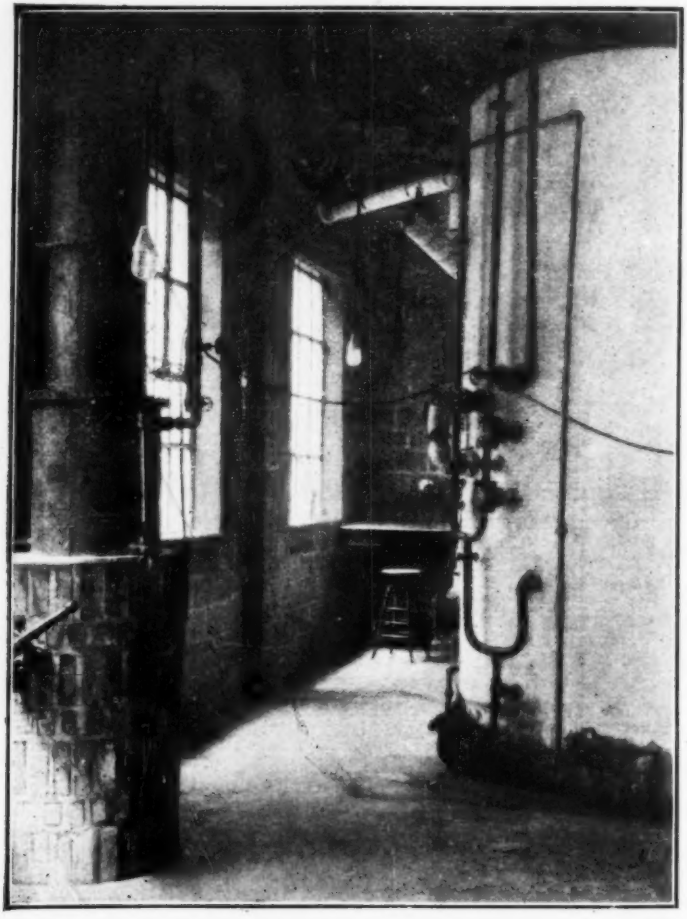
Discharging the extractor by a mechanical agitator theoretically appears to be worthwhile. An extractor of the proper size of 5,000 lbs. capacity can be discharged about as rapidly by hand. The danger of the agitator at this point is considerable. If the meal is improperly deodor-

Early form of Bataille Extractors Installed in England in 1892

ized, the solvent mixed with the air which enters when the discharging door is open, becomes an explosion hazard, since the agitator might strike a spark against a stray stone.

Size Units

Theoretically it should not be much more difficult to extract 50,000 lbs. at a batch than 5,000 lbs. It probably is not more difficult, but it is practically an impossibility to deodorize the residual meal. A large extraction plant for flaxseed was built in the United States some years ago which attempted to extract immense quantities in each charge. It was impossible to properly deodorize the meal and this unfortunate experience has probably hindered considerably the development of solvent extraction in the United States. Both the Bataille and the Scott extrac-

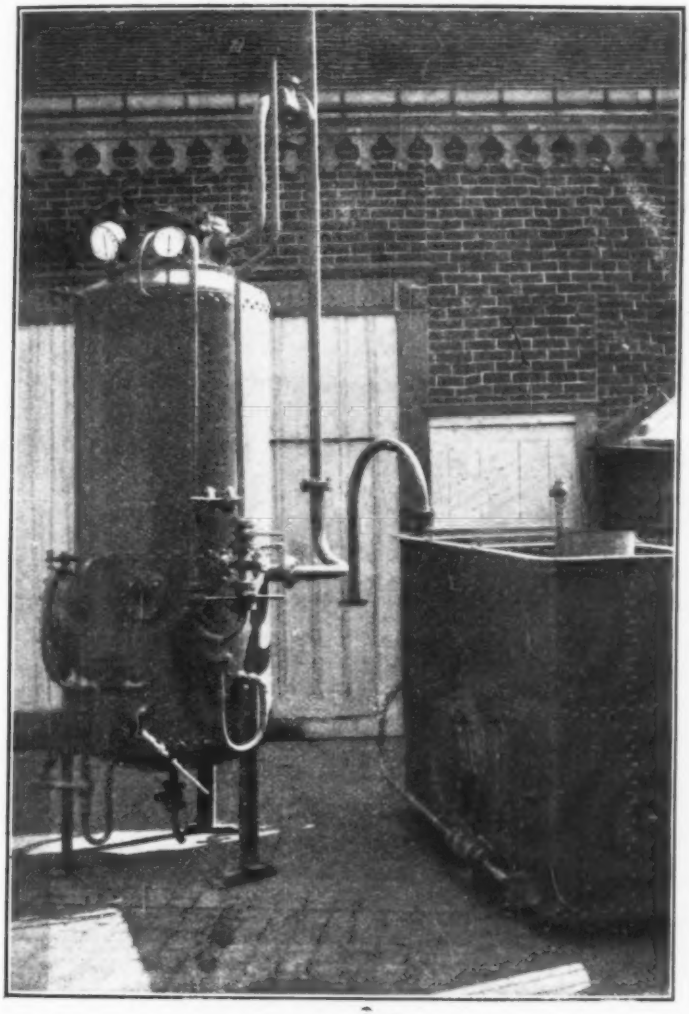


Modern form of Bataille Extractor without agitators, series flow of solvent or circulating pumps

tors are small. This is because the European experience upon which their designs are based has indicated that 5,000 lbs. or 6,000 lbs. capacity is the maximum size of which an extractor should be built.

Continuous and Series Extractors

The writer knows of only one installation of continuous extraction in this country. Since this plant is no longer in operation, he is under the impression that continuous extraction has not yet been fully developed.



Small extractor unit for Fuller's Earth. (Left) Combined Extractor and Still. (Right) Combined condenser and solvent reservoir, which is located above extractor when apparatus is assembled

It is doubtful whether it is feasible because the capacity per dollar invested is probably not as great as the intermittent extractor plant and the complications are manifold.

The semi-continuous or series extractor is an arrangement of 4 to 6 in a row with the solvent passing from one extractor to the next. Theoretically, this should save steam by producing a more concentrated oil solution. Practically, it is inadvisable because it means a complicated series of piping and circulating pumps handling hot solvent, both of which increase the solvent loss, which rapidly nullifies any steam saving. The greatest difficulty is the necessity of charging and dicharging all of the extractors in the series at the same time. This is evidently uneconomical and the practical result is that certain extractors are out of service part of the time awaiting their turn. In addition if difficulty is experienced with one extractor it holds up the rest of the family. Individual unit extractors, independent one from the other will be found to turn out a greater amount at the end of the year and operate more economically than the above arrangement.

Extraction Period

The extraction period is comparatively simple provided the extractor is kept filled with solvent and the proper arrangement of filters are used. The solvent that is generally employed in Europe is a cut of gasoline boiling at 60° C. with neither light nor heavy ends and which is available almost anywhere on the Continent. Trichlorethylene is also in general use because it is not inflammable. Benzol is excellent except that it frequently produces a darker oil. Carbon tetrachloride is not used because it is too easily hydrolized and attacks the metal container. Ethylene dichloride is similar to trichlorethylene except that the manufacturers claim that it hydrolizes less. It has the disadvantage of being slightly inflammable (about one-sixth as much as gasoline), and costs about the same price per pound as trichlorethylene.

In conclusion, the extractor which has no movable part, no pumps or agitators, which is of a proper size and independent in operation from any other extractor is the most practical type. It certainly is the type that most appeals to the oil plant operator who wishes to avoid the use of expensive technical supervision and undoubtedly in the next ten years many of the cotton oil plants will have adopted such an extractor and thereby recover sufficient oil to pay for the actual operation of their plants.

New York, N. Y.

OIL CONTENT OF VARIETY TESTS OF COTTON FOR 1924 AND 1925

The following table shows the per cent of oil in cottonseed and the gallons of oil per ton of cottonseed from the variety test of cotton at the Georgia Experiment Station for 1924:

Variety	Per Cent Oil in Cottonseed	Total Gals. Oil Per Ton of Cottonseed
Wannamaker's Cleveland	14.77	39.38
Coker's Cleveland, 1⅛	14.99	39.96
Coker's Piedmont Hartsville, No. 19	19.58	52.20
Acala	15.35	40.91
Webber's 49-101-3-3	18.63	49.67
Coker's Cleveland, 1⅛	14.36	38.28
Coker's Pedigreed Cleveland, No. 4	14.10	37.59
Poulnot	16.29	43.43
Durango	17.64	47.03
College No. 1	15.80	42.12
Delfos 631	17.47	46.57
Collin's Piedmont Cleveland	16.33	43.54
Piedmont Cleveland	15.71	41.88
Lone Star	16.23	43.27
Cook 588	16.61	44.28
Express 433	19.90	53.05
Addison's Extra Early	17.64	47.03
Trice	17.12	45.62
Deltatype	17.14	45.70
Wilkinson's Toole	15.89	42.36
Salisbury	18.55	49.45
Coker's Lightning Express	16.59	44.23
Steinheimer's Cleveland	16.47	43.91
Bouknight Cleveland	14.90	39.72
Delfos 6102	16.70	44.52

The following table shows the per cent of oil in cottonseed and the gallons of oil per ton of cottonseed from the variety test of cotton for 1925:

Variety	Per Cent Oil in Cootonseed	Total Gals. Oil Per Ton of Cottonseed
Latta	17.50	46.65
Delfos 631	18.41	49.18
Delfos 911	17.29	46.09
D. P. L., No. 4	18.29	48.76
Delfos 6102	17.84	47.56
Lone Star	16.25	43.32
Salsbury	17.02	45.37
Steinheimer's Cleveland	17.11	45.61
College No. 1	16.81	44.82
Deltatype Webber, Strain 4	18.13	48.33
Trice	16.80	44.79
Acala	17.82	47.51
Piedmont Cleveland	16.67	44.44
D. P. L. No. 5	18.81	50.15
Coker's Cleveland, Strain 4	15.35	40.92
Coker's Lightning Express	16.87	44.97
Collins' Cleveland	15.27	40.71
Wannamaker's Cleveland	15.35	40.92
Cook 588	17.95	47.85
Coker's Pedigreed Hartsville, No. 20	18.57	49.51
Coker's Pedigreed Webber 49	16.97	45.24
Coker's Cleveland	15.24	40.63

BULK CARGOES OF OIL

By H. P. TREVITHICK

In the past few years, the methods of importing vegetable oils, particularly into the port of New York, have been undergoing considerable changes.

Heretofore the usual method of shipment has been in barrels. Cottonseed oil, olive oil, olive oil foots or sulphur oil, soyabean oil, cocoanut oil, etc., have been shipped into the port of New York in this way. In some cases, new barrels were used; but with sulphur oil, second-hand barrels were generally to be found. Besides the barrels, however, pipes, puncheons, hogsheads, and casks are used. Palm oil, particularly, is shipped in casks containing about three-quarters of a ton of oil.

In all cases, these containers have to be handled many times. Thus, when filling, the barrels, etc., are rolled, coopered, etc. Then they must be transported to the ship, taken aboard by derricks, stowed away in the hold, and stand the strains of the voyage. Arriving on this side, the barrels must be unloaded, sampled, rolled, etc.

As a result of all this handling, many of the barrels leak and all must be coopered before they can be shipped to their destination. The charges for this may run as high as $1.00 or $2.00 per barrel in the case of second-hand barrels or poor shipments, and the delay to the shipment may run from a day or two to two or three weeks. In addition to this direct expense at the port of discharge, there is the cost of the barrels, the loss by leaking, the cost of delays in handling, etc. Moreover, if the barrels happened to get loose in the ship during a storm, anything may happen from the loss of the oil in a few barrels which may crush, to the general shifting of the cargo of a ship during rough weather and the possible damage to or loss of the craft.

In recent years, particularly since the great increase in the use of oil for fuel in ships, bulk shipments have been taking the place of barrel shipments. Ships have always had ballast tanks between the double bottoms, peak tanks in the ends of the ships, etc., which have been used for the storage of drinking water, and for "trimming" the ship, etc; but these are distinctly not suitable for the shipment of vegetable oils. The "peak" tanks are generally too deep, and narrow and obstructed for satisfactory discharge through their covers. The ballast tanks have no height, are full of obstructions from the frame of the ship and are too exposed to the cold temperature of the sea, particularly in winter. They are not more than 36 to 48 inches in depth, and run the full width of the ship, exposing such an enormous area to the cooling effects of the sea that steam coils are not of much use in melting the congealed oil. In one case, we have known of a 10 per cent loss of oil in such a shipment. Further, both of these kinds of

tanks are impossible to clean satisfactorily to prevent contamination.

With the advent of oil burners, it was necessary to get large tanks for fuel storage. These tanks are generally built just aft of the pilot house and forward of the boilers. There are generally two such "deep" tanks lying athwart the ship, one on each side, and separated only by the shaft tunnel and a partition above it. Each tank is approximately 20 to 25 feet deep, 20 feet in length, and half the width of the ship in width. They hold about 400 short tons each and are equipped with steam coils, bilges of their own, discharging pipes, and valves controlling them in the boiler room, and have plenty of room on the deck and large enough hatches, etc., to facilitate the charging and discharging of the cargo. They are primarily to be used for fuel storage, of course, but as this country is a source of oil supply, usually enough fuel is purchased here for a round trip. Then, on arrival on the other side, enough of the remaining oil is pumped into the peak tanks to supply the ship for its return voyage to this side and any surplus is sold. This frees both of the "deep" tanks for cargo. These are then cleaned with kerosene, steam, lye, etc., and the valves in the boiler room sealed or "blanked" off. They are then ready for cargo.

In the last few years, cocoanut oil, sulphur oil, palm kernel oil, palm oil, soyabean oil, and tung or Chinese wood oil have been received in such bulk shipments.

The advantages of the savings resulting from such shipments have been considerable. No barrels or other containers are required, so that their cost and the cost of filling is obviated. Then there is no other expense of loading except the actual cost of pumping from storage or railroad tank cars into the ship.

Of course, no loss by leaking should occur, and no shifting of cargo during transportation can occur. Thus any loss or damage is prevented not only to this shipment, but also any loss or damage to other cargo or to the ship itself.

On arrival, this oil can be pumped directly into tank cars brought alongside the ship on floats and transported direct to destination, without any delay, and with no losses or charges due to recoopering, etc. With a fair sized pump, such as one delivering 100 or 150 tons an hour, and with several flexible delivery lines so that 3 to 5 cars can be filled at one time, such a cargo of 800 tons can be discharged, including the cleaning of both tanks, in 24 to 36 hours. In barrels, this cargo would require over 4,000 barrels, and the incidental labor charges for handling, etc., would be considerable. If they needed much re-coopering the cost could easily be increased $4,000 to $1,000. The time saved is also considerable as the cargo can be discharged and shipped to destination in such a short time.

Of course, certain precautions must be observed. The oil must be hot enough to be thoroughly liquid when discharged. No contamination

should occur if the coils are tight and the tanks are thoroughly cleaned. When the bulk of the oil has been removed, it is necessary that men use brooms, squeegees, etc., to clean between the coils and to brush down the sides to get all the remaining oil into the bilges, whence it can be pumped. If the oil has not been kept liquid during the voyage, care must be taken in heating and melting or otherwise the oil around the coils will scorch and darken before the balance of the oil is thin enough to allow circulation.

In general, however, this method of shipping large quantities of oil results in great economy of time, money, trouble, etc.

NEW YORK PRODUCE EXCHANGE.
July 8, 1926.

P. S. TILSON

P. S. Tilson, one of the founders of the American Oil Chemists Society and a leading figure in the chemical profession and oil and fat industry, died on May 29 at the age of 59. For sometime previous he had been in poor health, and although he followed his custom of attending the May convention of the American Oil Chemists Society at New Orleans, he was unable to participate to any extent in the social diversions of that meeting. Shortly after his trip to New Orleans, he submitted to an operation and the week following he passed away.

Funeral services were held in Houston, from which place a great many friends accompanied the body to Bryan, Tex., where further services were held at the interment.

Mr. Tilson was graduated from Texas A. & M. college in 1888, and for some years after was connected with that College as instructor in Engineering Chemistry. In 1904 he moved to Houston and established the Houston Laboratories, of which he was the president at the time of his death. At one time he was a collaborating chemist of the U. S. Department of Agriculture, and later was Associate State Chemist for Texas. In addition to membership in the American Oil Chemists Society, he was a member of the American Chemical Society, the Interstate Cottonseed Crushers' Association, and the Texas Cottonseed Crushers' Association.

Mr. Tilson's death deprives the chemical profession and the oil and fat industry of a very dear friend and an associate whose character and attainments commanded the respect and confidence of all who knew him.

OPTICAL PROPERTIES OF LINSEED OIL

It is commonly known that modern pictures painted in oil were found to undergo a considerable lowering of tone after a few years. This went on over the whole surface, but it varied with different painters who followed their own particular methods. This state of affairs in relation to modern pictures was in strong contrast with pictures painted in the fifteenth century by Van Eyck and others, which to the present day retained their brilliancy of tone. Prior to the introduction of painting in oils the mediæval painters painted in tempera, but the paintings of the first painters in oils had produced works which retained their brilliance even to this day, as compared with the loss of tone in so many modern oil paintings.

Professor A. P. Laurie was one of a committee of artists and chemists appointed by the Royal Academy to investigate this and other problems affecting artists. Professor Laurie recently discussed some of the points under investigation at a meeting of the Oil and Colour Chemists Association in London. His paper entitled "The Optical Properties of Linseed Oil and the Technique of Van Eyck and his Followers" was ably reported by *Chemical Age*, London, and constitutes most interesting reading for the paint manufacturer and chemist.

This journal quotes Professor Laurie as stating that there is nothing in the historical information available concerning the art of the eleventh and twelfth centuries to suggest that there were any special methods of preparing the oil or pigment. The real fact was that Van Eyck and other painters of those days were first-class artists, and the suggestion that there was a tradition, which had grown up during 200 or 300 years from the eleventh or twelfth century onwards, was due more to picturesque writing than to actual fact. Therefore, the committee came to the conclusion that there was no mystery or secret about the methods adopted by the early painters and that the whole matter required looking into afresh.

Some Experiments

Professor Laurie said that he had tried a few simple experiments in order to get more information on the yellowing of oils used in the painting of pictures. He took artists' flake white and ground it in various oils, including artists' linseed oil which had been bleached in the sun, poppy oil supplied by a good firm, stand oil, copal varnish, etc., and from these experiments certain facts came out. The oil that altered least was the poppy oil; next to that was Dutch stand oil; next came walnut oil; and the one that yellowed most was linseed oil. Copal varnish yellowed more than any of the others, whilst turpentine yellowed worst of all.

It was rather an interesting fact that a great many artists held the view that when they used turpentine as a thinner the tendency was for the oil to yellow more than any other. Artists varnished their pictures with mastic,

and he took some of his white lead in oil grindings and put one sample on glass and another on an artist's canvas varnished with mastic. In yet another case he coated the back of the canvas as well as the front with mastic and found that the sample on glass was protected absolutely from the effect of moisture, the sample of canvas coated one side with mastic was fairly well protected, whilst the canvas coated on both sides with mastic was protected very much better. The canvas with mastic on both sides showed a little yellowing, the glass showed a little more, whilst the canvas with mastic on one side only showed the most yellowing of the three.

The important practical bearing of that from the artist's point of view was that when he varnished the front of his picture he did not protect it from moisture at the back, but it was due to the action of moisture or oxygen on certain ketones that yellowing took place and the more the picture was protected back and front the less it would yellow. Copal varnish yellowed considerably; stand oil in which dammar was dissolved, or linseed oil in which mastic was mixed, changed very little in tint and remained remarkably white. Therefore, he put the order as follows:— Poppy oil, stand oil in which dammar had been dissolved; linseed oil mixed with mastic; walnut oil; stand oil; finally linseed oil. These simple elementary experiments gave some practical information to the artist.

It was well known that when an artist painted out a portion of an oil painting, in the course of time the painted out part showed through. There were two explanations of this. One was the permeability of the oil in the pigment, and the other that saponification might be going on slowly between the white lead and the zinc oxide which produced this effect. There was, however, another possibility and that was whether the linseed oil film altered in refractive index. If that were so, it would account for a good deal of the lowering of the tone of oil paintings. Experiments were described which gave a rough classification of the pigments artists used in order of opacity, which might also be regarded as the order of refractive index. If a linseed oil increased in refractive index, as he claimed these experiments showed, then the pigment would be deeper in tone and the whole surface of the picture would be lower in tone. Thus the increase in the refractive index of linseed oil was an important matter in the lowering of the tone in oil paintings after a period of time, and he could not help thinking that in the increase of refractive index of linseed oil there would be found a solution of more than one trouble experienced by the painter in oils.

Discussion

The President, in thanking Professor Laurie for his lecture, said it had taken them back to the fundamental principles underlying some of the oil and color chemists' business. The present day oil and color chemist was rather more inclined to consider the problems connected with the manu-

facture of his materials than the scientific effects underlying the methods of application. It was only by realizing and studying these scientific effects that it was possible to manufacture what was required. He could personally substantiate the statement that oils mixed with turpentine showed a greater degree of yellowing than oils not so mixed.

Noel Heaton said that although Professor Laurie had stated that there was no mystery about the methods of the Van Eyck school, it was still a subject of tremendous controversy as to how those paintings retained their tone. The early painters were consummate craftsmen, and if work of this character was to be produced it needed the application of scientific technology of the highest degree. Whilst boiled linseed oil was known in the time of Van Eyck the painters of those days started their pictures with a foundation and built the whole thing up in a painstaking manner, which very few present day artists would take the trouble to do. That was the rock bottom secret of the whole matter. The modern artist simply squeezed his color out of a tube on to a brush and what happened to it afterwards he did not quite know.

Cruickshank Smith said it was interesting to hear that poppy oil compared so favorably with the others because in some experiments of his own certain types of stand oil were quite equal to poppy oil in their resistance to change of tint.

TAKES OVER HOUSTON LABORATORIES

F. R. Robertson has taken over the Houston Laboratories, Houston, Texas, following the death of its president and founder, P. S. Tilson. The work of the laboratory will be continued as formerly, under the direction of Mr. Robertson.

THE DISTILLATION OF COCOANUT OIL AT ESPECIALLY LOW PRESSURE

By H. I. WATERMAN and H. J. RIJKS*

Chevreul has already distilled not only fatty acids but also fats without decomposition in a Torricelliam vacuum[1]. Caldwell and Hurtley have distilled[2] butter-fat and cocoanut oil in a high vacuum and without decomposition. In the case of cocoanut oil almost 50 per cent is distilled up to 210° C. But their investigations are occupied more with fatty acids than with fats. Later on Bömer repeated the distillation of cocoanut oil[3]. He worked in a vacuum of the cathode light and obtained 87 per cent distillation at a temperature of 210-285° C. Bömer did not distill to completion, and moreover the residue underwent decomposition as distillation progressed, while in addition the distillate already obtained from redistillations under the same conditions exhibited decomposition phenomena.

We have made a further study of this question and have succeeded in completely distilling over cocoanut oil in vacuo without the occurrence of appreciable decomposition. This is made apparent by the constants obtained from analysis. But as far as smell and taste were concerned, the white fractions obtained showed no edible oil characteristics. The first fraction had a sharp (burning) taste and a somewhat pungent odor. This was caused by traces of decomposition products. It is also possible to re-distill the obtained fractions in vacuo.

The material forming the starting point of the tests consisted of 200 gr. of pure refined cocoanut oil obtained from one of the Dutch oil factories. There were obtained four fractions with a weight of 60, 35, 55, and 43 gr., or respectively 30, 17.5, 27.5, and 21.5 per cent. These fractions went over respectively at 206-223° C., 223-229, 229-231, and 231-254. The attenuation of the air during the distillation was not measured but throughout the entire experiment the Geissler tubes *J* and *X*, (see figure) remained unillumined, so that the pressure was too small to be measured by means of these tubes. Measurement of the temperature was carried out by means of a thermocouple; an ordinary thermometer inserted in place of the thermocouple *Q* was also used. The temperature at the point *R* was also capable of being determined by means of a thermocouple.

The figures obtained for the original cocoanut oil and for the distillates are given in the tables on the page following.

* Reprinted from the Journal of the German Oil and Fat Industry.

Refraction n_d (60° C.)

Original oil	1.4420
Fraction 1	1.4391
Fraction 2	1.4398
Fraction 3	1.4408
Fraction 4	1.4447

Acid number (Milligrams KOH per gram)

Original oil	0.945
Fraction 1	1.4
Fraction 2	0.9
Fraction 3	0.9
Fraction 4	0.27

Saponification number (Milligrams KOH per gram)

Original oil	256
Fraction 1	280
Fraction 2	274
Fraction 3	262
Fraction 4	237

Accuracy ± 2

Iodine number (Wijs)

Original oil	8.2
Fraction 1	2.4
Fraction 2	1.5
Fraction 3	4.3
Fraction 4	23.3

From these figures it appears that for practical purposes no decomposition of the fats into fatty acids has taken place.

If saponification numbers of the fractions obtained are considered additively, we find 25,600 per 100 gr. for the original oil;

for fraction 1	$30.0 \times 282 = 8{,}460$
for fraction 2	$17.5 \times 274 = 4{,}795$
for fraction 3	$27.5 \times 262 = 7{,}205$
for fraction 4	$21.5 \times 237 = 5{,}095$
Total	25,555

The losses during distillation were 3.5 per cent. In the fractions we find a total saponification number of 25,555—a figure of considerable size when considered in relation to the losses. In the same way we find a

small loss in the case of the acid number, a result which must be acknowledged as favorable.

The iodine number proves that those substances which fix iodine in Wijs's method are only recovered in the distillates of higher boiling point. This is in accordance with the observations of Bömer and Baumann.

The apparatus used will be found detailed in the figure cited. The vacuum was at first maintained by means of a preliminary pump I, aided by flasks containing carbon (H_1 and H_2), and placed in Dewar flasks containing liquid air, while at the same time a mercury pump G according to the design indicated by Langmuir[4] was shunted in.

From the distilling bomb A made of Pyrex glass and heated with the air bath P the fat was distilled. The distillate passed via D to the

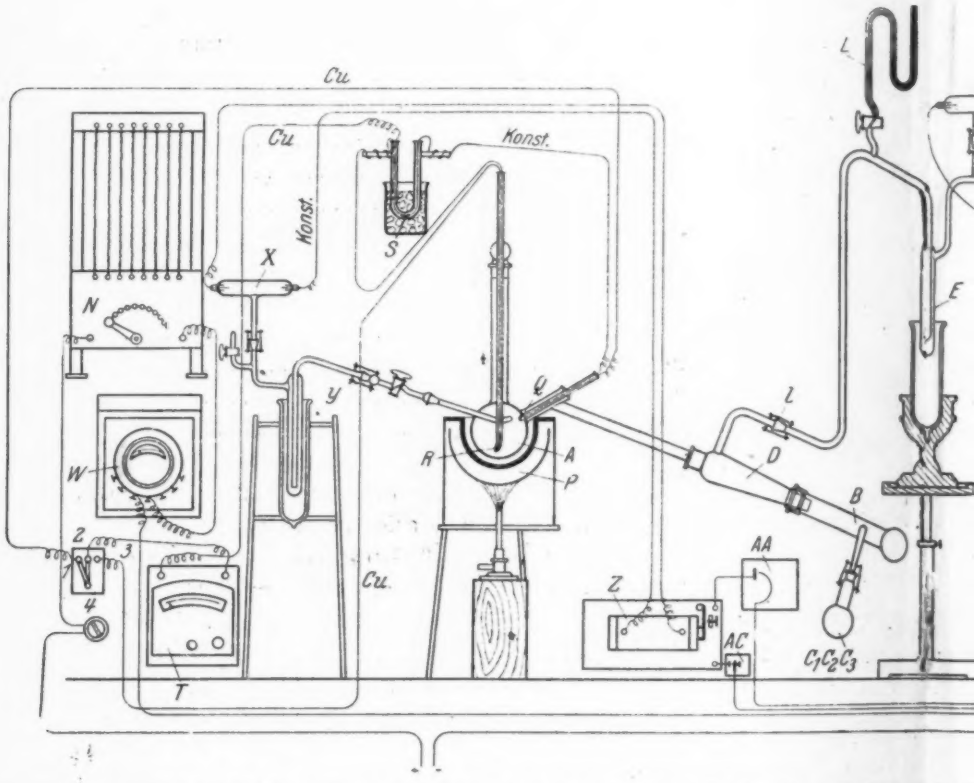


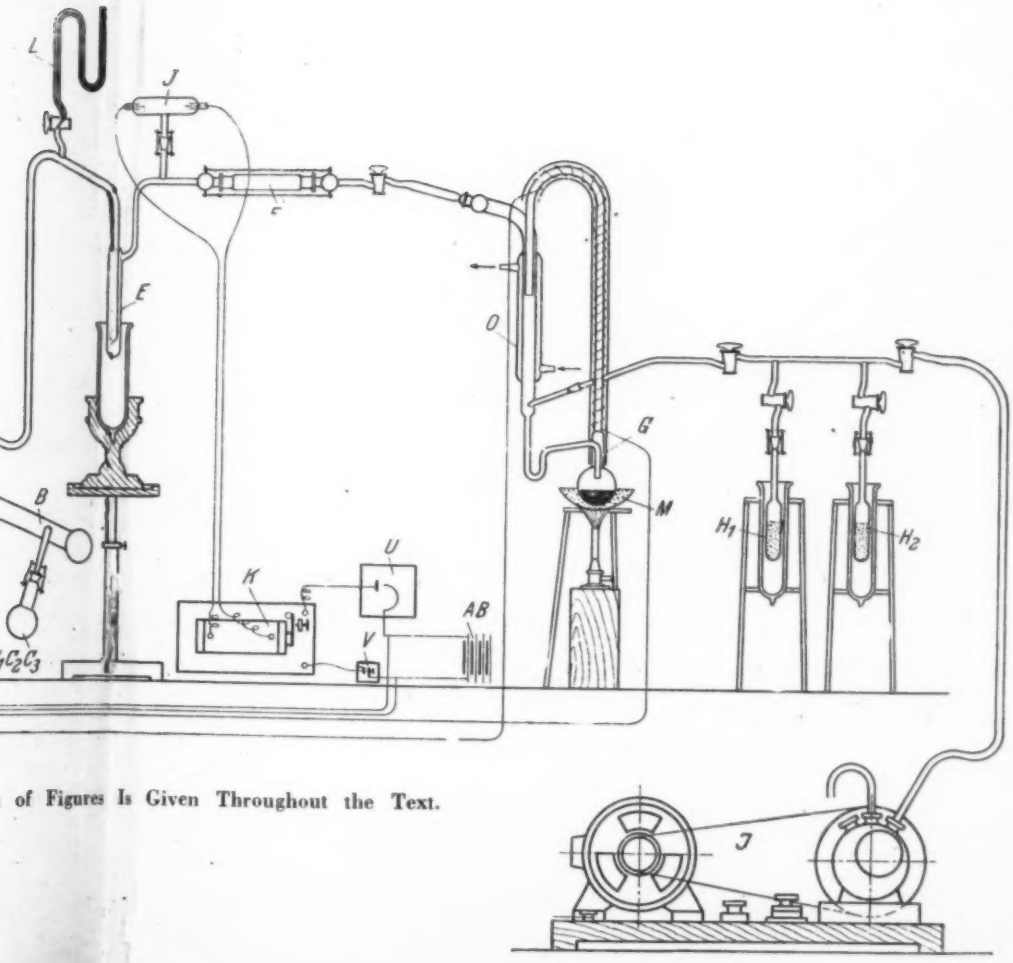
Diagram of Apparatus Used in the Study Reported in This Paper. Explanation of Figures Is G

receiver B equipped with the small bulbs connected by ground joints C_1, C_2, and C_3.

The space, which was highly evacuated, was made entirely of glass and so far as necessary, was fitted with carefully ground cocks and connecting pieces.

The vessel E (in a Dewar beaker containing liquid air) served to condense very volatile constituents which do not leave the receiver as condensates. In F there was placed a dish of P_2O_5.

The mercury pump was heated by means of the sand bath M. In order to prevent the condensation of mercury vapor in the riser, this tube was surrounded by asbestos wired on with nickel wire. This wire was connected with a direct-current circuit by means of a rheostat N. A cool-



of Figures Is Given Throughout the Text.

ing water connection served to cool the jacket *O* of the mercury pump.

We observed that during the distillation process (an undertaking of only a few hours) the liquid in the bomb *A* was entirely quiet[5] and did not exhibit the well-known "phenomena of boiling."

Because of the fact, already stated, that the pressure was too small to be measured even approximately by means of a Geissler tube passing an electric current, the distillation was repeated and in the course of this the vacuum was measured by means of a MacLeod manometer[6]. In this case also 200 gr. of cocoanut oil were distilled. The weights of the fractions recovered were respectively 26, 49, 71, and 47 gr. or respectively 13, 24.5, 35.5, and 23.5 or altogether 96.5 of the original product.

The temperatures at which these fractions distilled over (measurements at *Q*) were this time respectively 208 to 218° C., 218-223, 223-237, 237-259, measurements which were at the same time carried out with the ordinary thermometer.

The pressure at the beginning of the distillation was 0.0023 m.M. of mercury and at the end 0.0034, the vapor pressure of the mercury not being considered.

For the calculation of these pressures from the observed heights of the mercury column in the capillary tubes of the manometer we used the calibration values established for the manometer by Dr. G. Meyer, for which we sincerely thank him.

We are convinced that the ease with which distillations of this kind may be carried out can become of great importance for the analysis of fats. It is now possible to distill without decomposition those glycerides such as occur in cocoanut oil, and the analysis will undoubtedly be capable of being rendered more precise.

DELFT LABORATORY FOR CHEMICAL TECHNOLOGY OF THE TECHNICAL COLLEGE.

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1) Journal of the Chemical Society XCV, Trans. I page 853 (1909).
2) K. S. Caldwell and W. H. Hurtley, The distillation of butterfat, cocoanut oil, and their fatty acids, Journal of the Chemical Society, XCV, Trans. I page 853 (1909).
3) A. Bömer and J. Baumann, Contributions to the knowledge of glycerides of fats and oils IX. The glycerides of cocoa butter, Zeitschr. Unters. Nahr.-u. Genussmittel 40, 97 (1920). Cf. also Ch.-Ztg. 38, 844 (1914).
4) See for instance B. J. Heerdt, Overzicht van de theorie en de toepassingen van gassen, waarin de onderlinge botsingen der moleculen kunnen verwaarloosd worden, Utrecht-Nijmegen 1923, blz. 274.
5) Caldwell and Hurtley op. cit. page 855.
6) F. H. Newmann, The production and measurement of low pressures, London, 1925.
7) An apparatus from the collection of the laboratory for inorganic chemistry of the Technical College was kindly placed at our disposal.

THE ENZYME SPLITTING OF FATS

By Albert P. Sachs

The splitting of fats into glycerin and fatty acids is known in practical work as "saponification" which should more strictly be applied to the conversion of fats or fatty acids into soaps, i.e. the metallic salts of the fatty acids. The better term for the splitting of fats is "hydrolysis" and this is consistently used in scientific work. The chemical change which the fats (triglycerides) undergo on conversion into their hydrolysis products may be represented as follows:

$$C_3H_5\begin{matrix}OR\\OR'\\OR''\end{matrix} + 3MOH = C_3H_5(OH)_3 + \begin{cases}ROM\\R'OM\\R''OM\end{cases}$$

C_3H_5 (OR) (OR′) (OR″) represents a triglyceride in which the acidyl groups R, R′, R″ may be the same or different; MOH represents either water (HOH) or any metal hydroxid (NaOH or CaOH, where Ca = ½Ca). The products on the right hand are the glycerin formed and the free fatty acid or soap (or mixtures of free fatty acids or of soaps) formed.

The above equation is not necessarily that which actually occurs, but represents merely the end result; in the case of hydrolysis by steam under pressure it may and probably does represent the end result of several successive reactions, namely:

C_3H_5 (OR) (OR′) (OR″)+HOH=C_3H_5 (OR) (OR′) (OH)+R″OH

C_3H_5 (OR) (OR′) (OH)+HOH=C_3H_5 (OR) $(OH)_2$+R′OH

C_3H_5 (OR) $(OH)_2$+HOH=C_3H_5 $(OH)_3$+ROH

In discussing the enzyme hydrolysis of fats we shall touch on this reaction again.

Hydrolysis is usually carried out by one of the following methods, which are by no means of equal technological value and importance. Thus for instance, hydrolysis by ammonia under pressure, is theoretically very intriguing because the ammonia soap which is formed can be decomposed into free fatty acid and ammonia by blowing steam through the soap; all the ammonia is recoverable. On paper we have thus an effective means of hydrolyzing fats into free fatty acids and glycerin without using up any chemicals at all, but the process is apparently not commercially feasible.

The methods are:

1. By steam alone at elevated temperatures; fifteen atmospheres pressure and 200° C. are necessary, and the product is darkened.

2. By means of steam and lime in open kettles (i.e. not under pressure nor at elevated temperatures). About 9.7 per cent actual lime, CaO, is theoretically necessary, but in practise 12-14 per cent of lime must be used to obtain complete saponification.

3. By means of steam at moderately elevated temperatures and a

catalyst or accelerator. Examples are steam at eight atmospheres with 3 per cent of lime or at twelve atmospheres with 1 per cent of lime. Saponification, i.e. the formation of calcium soaps is complete to the extent of the lime present, but free fatty acids are also present due to the insufficient lime present to combine with all the fatty acids. Magnesia or zinc oxide with or without zinc dust are also used, as is lime with zinc oxide or zinc dust, which seems to diminish discoloration.

4. By means of excess alkali in open heated vessels. A variant of this is the use in the laboratory of alcoholic caustic potash or caustic soda for saponifying small samples of fat completely.

5. By ammonia under pressure with subsequent removal of ammonia by blowing out with steam.

6. By acids. The use of muriatic acid (HCl) is not commercial, but that of 4-6 per cent of 66-67°Bé sulfuric acid (H_2SO_4) is commercial.

7. By Twitchell's reagent and similar hydrocarbon-oleic sulfonic acid reagents, etc.

8. By enzymes (also called "ferments").

The water hydrolysis, open kettle lime process and the ammonia processes are not used commercially; all the others find some use in commercial plants, though by no means to the same extent. The enzyme splitting of fats is one of the less important commercial methods but is nevertheless extremely interesting and is subject to possible improvements which may greatly increase its use.

Much work had previously been done on a scientific basis in studying the lipolytic (fat-splitting) action of certain ferments or enzymes, but without any attempt or indeed possibility of applying the results to technical purposes when Connstein, Hoyer and Wartenberg (Ber. d. deut. chem. Ges. 1902, vol. 35, pp. 3988-4006) reported their work. They stated that by using the enzyme present in castor beans (merely by grinding up the beans) fats could be hydrolyzed so effectively that the fatty acids yielded a pure white stearin directly by chilling and pressing while the olein was lighter than the commercial olein available; the glycerin was obtained directly in 40-50 per cent concentration with a small quantity of salts and some albuminous substances and peptones formed from the albuminoid substances by the peptonizing ferment present. The organic impurities could be removed by bone-black. The actual commercial results have proved by experience to be neither so favorable nor so simple as the first laboratory results seemed to indicate.

Let us for a moment consider the problem of the nature of an enzyme. Enzymes are catalysts produced by living matter (i.e. the lipase or fat-splitting enzyme of the castor bean). No enzyme has been prepared in a state of purity. Practically all enzymes are colloids or are intimately associated with substances having colloidal properties. Enzymes are generally

ssociated with protein matter either as an essential part of the protein nolecule or accompanying it. (See K. G. Falk "The Chemistry of Enzyme Actions," Chapter VII.) We know enzymes by their effects and not by their constitution.

Castor beans contain a lipase. At first ground castor beans (including husks) were used but technical difficulties arose, especially the low grade glycerin produced. Decorticating the seeds helped somewhat as the husks do not contain any enzyme but the trouble was not removed.

Nicloux (French patent 335902 of Nov. 14, 1903) introduced an improvement by triturating decorticated castor beans with castor oil or cotton seed oil; the product was filtered and the turbid oily filtrate centrifuged, yielding three layers. There is the excess oil on top, the practically inactive aleurone (starch) grains and membranes at the bottom, while the center active layer contains the cytoplasm (active cellular material) emulsified in oil. Nicloux used magnesium and calcium salts as activators of his ferment preparation (Nicloux's monograph, "Contribution á l'etude de la saponification des corps gras," Paris 1906, contains all the work done up to that time) but since then manganous sulfate has been found to be a more effective activator.

Nicloux's ferment-oil has since then been replaced by a "ferment-milk" due to Hoyer and first described by him in 1907 (Uber fermentative Fettspaltung, Hoppe-Seyler's Ztschr, f. phys. Chem. 1907, vol. 50, p. 1429). Decorticated castor beans are pulped with water in a suitable mill. The resultant emulsion is filtered from coarse suspended matter and the white creamy liquor is permitted to ferment spontaneously at about 24°C. Water separates and a thick creamy layer rises to the top. This is the active material and contains 38 per cent castor oil fatty acids, 58 per cent water and 4 per cent albuminoid substances including the actual lipolytic agent.

As the enzyme is practically insoluble in the oil, present practise is to cold press the seeds first to obtain the valuable first cold pressed castor oil and to work up the press cake for ferment-milk.

This ferment-milk is used as follows: In a lead lined iron tank the liquid fat is stirred up with about 35 per cent of its weight of water, 5-8 per cent. of "ferment" (i.e., "ferment-milk") and 0.2 per cent of manganese sulfate. The preferred working temperature is about 25°C. Once established this is generally maintained especially as the process liberates enough heat to raise the temperature spontaneously 2-3°C. Stirring by air is carried out from time to time in the early stages of the process to prevent separation of the emulsion, but towards the end the emulsion remains permanent. When the desired degree of hydrolysis is obtained (about 80 per cent after 1 day, 90 per cent after 2 days) the temperature is raised to 80-85°C. to kill the enzyme, 0.2-0.3 per cent (on weight of fat) of 66° Bé. sulfuric acid is added and the mixture stirred by air. On permitting to settle three layers form:

1. Aqueous glycerin (12-25 per cent glycerin) at bottom. This contains manganous sulfate, sulfuric acid, water soluble fatty acids and albuminoids.
2. Clear fatty acid layer on top.
3. Middle layer consisting of emulsion of albuminoids, glycerin, fatty material and water.

Several notes may be made at this point on the process. The cytoplasma in contact with water begins to be destroyed at 43°C. Hence the process cannot be carried out above 41° or 42°C. The fat must be liquid; therefore, high-melting fats cannot be hydrolyzed by the enzyme, unless they are admixed with sufficient low melting fats to remain liquid at about 35°C. It is best to operate at least 2-3°C. above the melting point of the fat and the best working temperature is about 25°C. Tallow is difficult to work with. Even with low melting tallows it is difficult to obtain above 75 per cent free fatty acids.

The enzyme works best in a very dilute acid solution. The free fatty acids produced regulate the acidity.

The amount of water used may vary from 30-40 per cent of the weight of the fat. Less than 30 per cent can be used only for fats with a high free fatty acid content such as solvent extracted bone-oil and peanut oil. More than 40 per cent of water dilutes the glycerin unnecessarily.

The action of the enzyme is reversible. If fatty acids and 96 per cent glycerin are mixed and the enzyme added, neutral fat will be produced. Welter (Zeitschr. f. angew. Chem. 1911, vol. 24, p. 385) obtained in this manner 35 per cent of neutral fat from palm kernel oil fatty acids and glycerin. It has been found that the reaction in hydrolysis by enzymes is not stepwise but is represented by the first equation of this article.

The glycerin obtained is generally considered superior to that from the Twitchell process or the autoclave process. However, Contardi (Giorn. di Chimica ind. ed appl. 1925, vol. VII, No. 6) who has had experience with the enzyme process at the Sirio plant at Milano-Bovisa, Italy, considers enzyme glycerin as of low quality due to the nitrogenous substances dissolved in the glycerin which cause distillation difficulties.

The enzyme process simple as it seems requires a careful technic. Slight changes in conditions produce great differences in results. Contardi gives a striking example of the limitations of our knowledge of the behavior of fat splitting enzymes. The subterranean vaults of the Rotunda at Milan served as the cemetery of the Milan Municipal Hospital and are estimated to have received 150,000 corpses from 1698 to 1783. These vaults were cleared out in 1907-1909. About one-fourth of the corpses, namely part of those in the east crypt had not undergone ordinary disintegration in their rest of more than a century in this vault. Owing to the absence of oxygen, presence of moisture, traces of ammonia, the uniform and moderate temperature, the steapsins (fat-splitting enzymes

present in animals) of the body had met such conditions that they converted all the fat of the tissues to free fatty acids before ordinary decomposition took place and these fatty acids remained unchanged preserving the form of the body and even the expression of the face. After opening of the vault and removal of the bodies disintegration set in immediately. This gruesome incident serves merely to show the undiscovered possibilities of enzyme action under suitable conditions.

Baur proposed the use of jancreas enzyme for fat-splitting. This was not commercially successful, and as the pancreas is our present source of insulin it is unlikely that pancreatic enzymes will be used for commercial fat splitting.

The glycerin obtained, in spite of certain obvious disadvantages, is free from trimethylene glycol. To purify the glycerin it is treated at the boiling point with milk of lime which precipitates manganous sulfate, sulfuric and fatty acids and some nitrogen compounds. After filtration a slight excess of sulfuric acid is added to remove lime. On concentration this glycerin is saturated with calcium sulfate. If this is undesirable the careful limed glycerin is treated with barium hydroxide to remove sulfates and then with oxalic acid to remove lime. On concentration by evaporation in vacuum a commercial glycerin is obtained with 0.2-0.5 per cent ash, and 0.5-1.2 per cent organic impurities. It can be easily purified by distillation.

The chief disadvantage of the process is the formation of the middle layer. These "middles" are collected, rewarmed and diluted and generally a further separation into three layers occurs. If run in connection with a soap factory this material can be run into the soap kettle and worked up with the usual run of fats.

A diagram of an enzyme installation is given in vol. 5 p. 446 of Ullmann's Enzyklopaedie der technischen Chemie, while a whole plant layout for castor bean handling down to the production of fatty acids and glycerin is given on a 1:125 scale in F. Goldschmidt "Chemie, Analyse, Technologie der Fettsäuren, des Glyzerins, etc.," vol. 2, page 54.

The costs for enzyme splitting are not known exactly but for comparative purposes it may be stated that Ullmann gives 3 marks per 100 kilos ($6.49 per ton) of fat hydrolyzed by enzymes, while he gives 1½ marks ($3.25 per ton) for the Twitchell method.

The advantages of the enzyme method are:

1. Light color of fatty acids.

2. Operation at lower temperatures with resultant less wear on equipment and lower amortization and repair charges.

The disadvantage is the formation of the special middle layer.

The following table of dydrolysis tests made b ythe Vereinigte Chemi-

schen Werke A.-G. of Charlottenburg, Germany, who operated the first enzyme splitting plant is self-explanatory.

Type of Oil Used	Weight in Kgs.	Water, kgs.	% on oil	$MnSO_4$, kgs.	% on oil	Ferment, kgs.	% on oil	% Hydrolysis	Fatty Acids, kgs.	Glycerin Anhydr., kgs.	% on oil	Middle Layer			
												Wt. Kgs.	% on oil	Fatty Acids	% on oil
Linseed, crude.	877	307	35	1.75	0.2	53	6	90	858	79.7	9.1	26	3.0	6	1.0
Linseed bleached	532	212	40	1.00	0.2	26.5	5	91	511	50.3	9.7	17	3.0	7	1.0
Cottonseed,															
American	900	360	40	1.80	0.2	54	6	86	870.5	80.5	8.94	34	3.9	14	1.6
English	912	364	40	1.80	0.2	55	6	91	888	82.1	9.0	42	4.5	8	0.9
	780	276	35	1.52	0.2	53	7	89	760	70.2	9.0	24	3.0	7	1.0
Tallow	904	326	35	1.90	0.2	90	10	86	888	78.5	8.7	40	4.4	16	1.8
Palm Kernel	845	338	40	1.70	0.2	68	8	84	833	85.1	10.1	19	2.0	6	0.7
Palm Kernel	816	326	40	1.63	0.2	65	8	90	795	84.3	10.3	28	3.0	8	1.0
Totals and avgs.	6566	2509	38.6	13.1	0.2	46.4	7	88.4	6403.5	610.7	9.4	230	3.5	72	1.1

U. S. ATE 5,000,000,000 POUNDS OF FAT IN 1925

In 1925 over five billion pounds of various kinds of edible fats were consumed by the people of the United States, according to a statement issued by F. M. Barnes, head of the Edible Oils and Fats Division of the Procter and Gamble Company. This is an average of 46.1 pounds per capita, which is an increase of one-fifth pounds per capita over the 1924 figures.

Another interesting indication of America's dietary habits is the swing to vegetable fats. Twenty-five years ago, lard was practically the only cooking fat used in the home, whereas now, according to government figures, vegetable fats make up thirty-four per cent of the actual amount used in American kitchens.

CO-OPERATION IN THE INDUSTRY

By W. H. JASSAPON

I am informed by your President that this is the first time a layman of the Industry has ever spoken before The American Oil Chemists' Society. This emphasizes the lack of co-operation which has kept our industry from making the progr it should.

I want to plead for mo co-operation between the laboratory and the office. I desire to enlist your interest to bring this about. You are the vital link between the seed pile and a profit account. The whole trouble has been that we have only looked upon your service in a most perfunctory way. I firmly believe if not for the fact that we sell meal and oil on analysis, much less use of your facilities than is even now the case would be demanded. And that feature of your work should be the small end of it.

I read recently where a large corporation stockholder switched many of his investments before leaving on a protracted trip, on the theory that he did not want to own common stocks in companies which had not research departments. He might find on his return that some of his investments were of little value because we were revolutionizing methods and practices so rapidly in this day and time that progressive institutions might be those who went along in a set manner out of business. Now this is true. And is this not one reason why the cotton oil industry has not attracted outside capital? Our methods and practices are too antiquated.

Personally, I believe the average crusher does not understand what the laboratory can do for him. He does not realize you can tell him the kind of raw material he is buying, and, what he can expect to make out of it, as well as what he is obtaining. Even then, many do not know now how to use such laboratory information. Your province is, therefore, one of education. I have held to the theory that one reason for the erratic competition in the purchase of our raw material is this fundamental lack of knowledge in many instances of what the probable value of the cotton seed really is. By a mere progressive application of your efforts, you can be instrumental in aiding to correct this unsound condition.

I do not want to take your time from pressing duties to listen to all that might be said on the subject I am discussing, but I do ask your indulgence for just a few more minutes to offer sincerely some suggestions which I hope your society will carefully consider.

In the first place, the industry should have a laboratory clearing house. In connection with the study which has been made by Mr. Meloy of the Department of Agriculture, looking toward a practical plan for grading cottonseed, he has been handicapped by a lack of information, which only the Laboratory can supply. Frankly, we really know little about our own business. What profits we may have made have been more

largely the result of good market guessing, other than from sound knowledge and intensive manufacturing application. We can never eliminate the hazard of industry, but by greater knowledge of what we are doing, we can largely diminish our risk. I have seen enough results from different mills to tell you that we are throwing away more money per ton of seed in our operations than we are making by watching the markets. The trouble about market guessing is that we cannot do it correctly consistently. We win some years, and we lose others, and when we lose, it is usually greater than our previous winning. Therefore, mills are continually changing ownerships, new faces take the old places and we start over again on the same road which leads nowhere.

Your services will find increasing demand if you make them more valuable. I hold it is not sufficient to simply analyze a sample. You should call attention to any point which a particular analysis may indicate. This is the principle of service. I had a large operator tell me recently he had no faith in analyses of cottonseed; he did not believe the low yield certain seed analyses showed. He said he could tell from the flow of the presses he was getting more oil! Now, this is what we have to overcome. We must quit guessing, we must find out, and you must help us find out the facts.

You should see that analyses are uniformly made and the percentages stated on the same basis. Some of you have discovered this year that some seed which cut perfectly prime, under present Interstate rules, made off oil. So far, none of the commercial laboratories have ever tested regularly the fatty acid in seed, and no provision has been made for deductions to compensate the mills for probable allowances they will have to make. Also very few of you test the percentage of meats in seed. Many chemists do not show the percentage of dirt and foreign matter, and I am sure that many mills do not know how to figure their yields against seed analyses.

I, therefore, again urge upon you the advisability of standardizing your analyses, and show thereon the information which the mills should have in order to more intelligently buy their seed. Some of you may argue that this will increase the cost of such complete analyses. Speaking for myself, I am not interested in the cost as much as I am in knowing what I am doing, but I will further add that by making your services so valuable, you will so increase the volume of your work, it should be possible even for you to reduce the cost.

Mr. Meloy will talk to you about the proposed plan for grading cottonseed, the first step in our endeavor to place the buying of our raw material on a more equitable basis, not only to ourselves but to the producer. Please remember it is only a suggestion. He needs and desires your interest and constructive aid, which I most earnestly ask you to contribute. It is the most far reaching and progressive step the industry has ever con-

templated. If Mr. Meloy's plan is a practical one, we want it and we will find the machinery to put it into effect. If you have a better scheme, submit it, and let us put it to the same searching analysis. We must find some simple and practical method to grade cottonseed. Practically all other farm commodities are sold on grades, and the cottonseed crop is too valuable to be handled any longer in the crude way we have been following. I have suggested to the Interstate Association that a cash prize be offered for a practical sampling tool and a quick and accurate method of separation of meats from hulls. I know we have the ability to work out these problems, once we set ourselves to the need for it.

Just one thought more—I would like to see the Interstate Association in co-operation with your organization, work out a definite plan for a research laboratory. It cannot, it should not be done by individual companies. We know all too little about extraction, about seed cleaning—in fact, about every single operation in an oil mill. But we will never make the progress which is possible until we scientifically and systematically set out to study these problems, in the abstract, and unhampered by immediate results.

CENSUS OF CORN OIL MANUFACTURES

The Department of Commerce announces that, according to data collected at the biennial census of manufactures, 1925, the establishments engaged primarily in the manufacture of corn sirup, corn oil, and starch in that year reported products valued at $132,873,000. Among these products were 101,641,000 pounds of corn oil, valued at $14,093,000; 33,300 tons of corn-oil cake and meal, valued at $1,317,000; 522,000 tons of stock feed, valued at $17,934,000; and other products, valued at $3,849,000.

The total value of products, $132,873,000, shows an increase of 14 per cent over $116,560,000 in 1923, the last preceding census year. In some cases the values of the several classes of products increased while the quantities decreased, and in others the values increased at higher rates than the quantities. The quantity of corn oil decreased 6.4 per cent and the value increased 1.6 per cent.

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RULE 270-A IS REVISED

At the annual spring meeting of the American Oil Chemists Society, a recommendation was made by the Seed Analysis Committee to the effect that Rule 270-a of Methods of Chemical Analysis be amended. The revision is in the form of a new section added for the purpose of defining a method for determining the oil and ammonia content of cottenseed. The new section is based on the Malowan method which has been in general use for a number of years, and modifications of the method recently made by C. H. Cox. The entire proceedure as recommended to the Society and adopted by it now constitutes Section 2 of the rule, and reads as follows:

Sec. 2. Determination of Oil and Ammonia in Cottonseed.

(a) Apparatus.

Oven. Well ventilated and capable of quickly reaching and maintaining a temperature between 120 to 130 deg. C. Heated by gas or electricity.

Vessel. A porous earthenware vessel, such as a 2½ inch flower pot capable of holding 50-60 grams of seed.

Pipette or burette to measure concentrated hydrochloric acid.

Grinding Mill. A mill with plates suitable for grinding treated seed to pass 30-mesh sieve.

(b) Process.

Absorb into the inner walls and bottom of the flower pot 1.5-2.0 cc. concentrated hydrochloric acid, then loosely fill the pot with 50-60 grams of cleaned seed.

Place in oven heated to 120-130 deg. C. and maintain at this temperature for one hour or until the seed are dry enough to grind well. When seed are properly treated they do not have a burned or charred appearance but the lint is brittle and disintegrates completely on grinding so that the ground seed is free from any appearance of lint or fibre and contain from 3.7% moisture.

When seed are removed from the oven they are cooled to room temperature and ground on any suitable mill to pass a 30-mesh sieve.

Analysis of the ground material is made by the Official Method for Moisture, Oil and Ammonia in cottonseed meal, except that the oil determination is given a four (4) hour extraction. It is suggested that the usual weight be used for ammonia; 4-5 grams for oil, and 5 grams for moisture.

(c) Calculation of Analysis.

A moisture determination is made by the Official Method on the original sample of seed at the time of receipt and the figures obtained for oil

and ammonia on the partially dried sample calculated back to the original basis by a factor derived from the following formula:

M equals Moisture in original seed.

P equals Moisture in partially dried seed.

F equals Factor to multiply by to reduce to original moisture basis.

(100 minus M) divided by (100 minus P) equals F.

It is suggested that the percentage of available ammonia in the seed be reported by subtracting 0.2 from the total percentage, this being the approximate average percentage of ammonia in the seed that is lost in the hulls produced.

It is also suggested that for the sake of uniformity all laboratories calculate "Available Oil" by subtracting from the total oil a weight of oil based on a standard of 80 (Ammonia multipied by .8 equals Oil), as applied on the weight of cake obtained from the "Available Ammonia" in the seed.

Example: Seed containing 3.75% Available Ammonia and 19.00% Oil and calculation is based on making 937 pounds of 8% Ammonia cake. A standard of 80 is equivalent to 6.40% oil in cake or 60 pounds. The available oil, clean seed basis, is therefore 320 pounds.

STATISTICS OF FATS AND OILS

The Department of Commerce announces that the factory production of fats and oils (exclusive of refined oils and derivatives) during the three-month period ended March 31, 1926, was as follows: Vegetable oils, 876,905,865 pounds; fish oils, 7,376,535 pounds; animal fats, 578,252,552, pounds, and grease, 88,800,914 pounds; a total of 1,551,335,866 pounds. Of the several kinds of fats and oils covered by this inquiry, the greatest production 567,614,937 pounds, appears for cottonseed oil. Next in order is lard with 462,365,154 pounds; linseed oil with 194,607,038 pounds; tallow with 113,509,554 pounds; coconut oil with 63,088,814 pounds; and corn oil with 33,929,759 pounds.

The production of refined oils during the period was as follows: Cottonseed, 500,921,044 pounds; coconut, 47,406,521 pounds; peanut, 2,170,164 pounds; corn, 25,206,201 pounds; and palm-kernel, 2,067,437 pounds. The quantity of crude oil used in the production of each of these refined oils is included in the figures of crude consumed.

The data for the factory production, factory consumption, imports, exports and factory and warehouse stocks of fats and oils and for the raw materials used in the production of vegetable oils for the three-month period appear in the following statements:

Production, Consumption, and Stocks of Fats and Oils

(In some cases, where products were made by a continuous process, the intermediate products were not reported.)

Kind	Factory operations for the quarter ending March 31, 1926 Production (pounds)	Consumption (pounds)	Factory and Warehouse Stocks March 31, 1926 (pounds)
VEGETABLE OILS[1]			
Cottonseed, crude	567,614,937	592,700,391	90,620,925
Cottonseed, refined	500,921,044	318,670,306	303,678,337
Peanut, virgin and crude	4,000,808	2,791,179	1,115,134
Peanut, refined	2,170,164	2,244,159	1,179,058
Coconut, or copra, crude	63,088,814	87,039,268	62,692,374
Cocoanut, or copra, refined	47,406,521	45,877,228	12,697,563
Corn, crude	33,929,759	32,035,355	10,266,368
Corn, refined	25,206,201	4,107,166	12,031,492
Soya-bean, crude	990,464	1,377,911	5,537,774
Soya-bean, refined	.....	1,389,749	1,052,115
Olive, edible	1,011,218	293,944	8,067,946
Olive, inedible	17,840	2,457,224	3,375,818
Sulphur oil, or olive foots	.....	8,372,503	5,204,280
Palm-kernel, crude	.....	24,212,692	25,342,941
Palm-kernel, refined	2,067,437	4,090,186	276,806
Rapeseed	73,100	3,250,293	4,589,237
Linseed	194,607,038	98,904,529	190,421,194
Chinese wood or tung	.....	20,449,055	35,822,084
Chinese vegetable tallow	.....	514,368	50,480
Castor	11,209,205	4,819,233	6,067,169
Palm	.....	29,563,593	31,373,958
All other	362,682	1,844,388	4,168,830

FISH OILS: (1)			
Cod and cod-liver	131,355	3,088,875	4,284,966
Menhaden	.....	11,319,094	17,059,000
Whale	.....	13,149,187	8,676,030
Herring, including sardine	7,100,640	4,333,637	4,018,924
Sperm	.....	327,004	769,362
All other, (including marine animal)	144,540	256,382	648,099
ANIMAL FATS:			
Lard, neutral	15,913,449	6,544,694	6,282,858
Lard, other edible	446,451,705	3,353,936	95,134,637
Tallow edible	13,529,544	9,184,851	4,467,188
Tallow, inedible	99,980,010	106,480,592	79,235,769
Neat's-foot oil	2,377,844	1,423,361	1,384,688
GREASES:			
White	20,943,721	8,200,274	8,685,316
Yellow	16,886,299	22,092,725	8,710,467
Brown	8,950,926	7,673,657	6,188,283
Bone	4,293,941	672,251	1,005,248
Tankage	13,066,913	622,170	2,959,322
Garbage or house	16,956,326	22,205,745	8,647,047
Wool	1,319,970	1,391,933	2,418,200
Recovered	3,752,615	1,680,917	1,041,879
All others	2,630,203	1,437,690	2,126,817
OTHER PRODUCTS:			
Lard compounds and other lard substitutes	288,784,907	72,065	16,660,177
Hydrogenated oils	102,521,486	101,054,686	16,855,551
Stearin, vegetable	3,679,204	6,106,406	1,829,649
Stearin, animal, edible	20,628,523	13,077,755	8,299,069
Stearin, animal, inedible	8,413,300	4,663,710	2,955,129
Oleo oil	40,616,480	11,772,055	11,541,871
Lard oil	9,474,469	4,997,232	4,353,208
Tallow oil	2,594,485	2,280,051	1,830,724
Fatty acids	30,907,333	28,158,058	3,959,501
Fatty acids, distilled	13,785,942	14,767,948	2,069,388
Red oil	12,202,776	7,136,716	7,844,188
Stearic acid	8,215,189	2,630,139	3,589,829
Glycerin, crude 80% basis	28,903,966	33,487,739	7,663,462
Glycerin, dynamite	10,543,997	8,905,996	7,076,646
Glycerin, chemically pure	16,436,489	1,568,459	4,243,421
Cottonseed foots, 50% basis	170,327,343	117,154,135	42,134,095
Cottonseed foots, distilled	35,057,784	35,981,013	13,819,637
Other vegetable oil foots	9,135,855	6,228,070	2,637,863
Other vegetable oil foots, distilled	447,419	265,268	466,721
Acidulated soap stock	64,040,871	21,449,662	46,223,574
Miscellaneous soap stock	4,217,130	2,888,479	4,475,350

Raw Materials Used in the Manufacture of Vegetable Oils—Tons of 2,000 lbs.

Kind	Consumed January 1 to Mar.31	On hand Mar. 31	Kind	Consumed January 1 to Mar.31	On hand Mar. 31
Cottonseed	1,959,462	415,277	Corn germs	64,880	589
Peanuts	4,578	1,290	Flaxseed	298,231	97,227
Peanuts in the hull.	2,073	135	Castor beans	12,904	4,485
Copra	48,520	6,225	Mustard seed	327	936
Coconuts and skins	1,033	97	Soya-beans	3,873	1,980
Rape seed	160	30	Olives	3,207	30
			Other kinds	334	757

[1] The data of oils produced, consumed and on hand by fish oil producers and fish canners were collected by the Bureau of Fisheries.

Imports of Foreign Fats and Oils, Quarter Ended Mar. 31, 1926

Kind	Pounds	Kind	Pounds
Whale oil	2,577,982	Palm-kernel oil	34,440,753
Cod and cod-liver oil	4,854,120	Peanut oil	836,769
Other fish oils	4,594,155	Rape oil	6,199,035
Beef and hog fats	8,053,769	Linseed oil	4,272,062
Wool grease	3,823,503	Sessame oil	1,516,056
Grease & oils, n.e.s. (value)	111,496	Soya-bean oil	6,010,101
Chinese wood oil or nut oil	23,754,435	Vegetable tallow	469,218
Coconut oil	54,666,396	Vegetable wax	2,217,511
Olive oil, edible	17,478,937	Other vegetable oils	4,790,296
Sulphur oil, or olive foots	9,985,327	Glycerin, crude	8,775,533
Olive oil, denatured	3,201,176	Glycerin, refined	1,669,353
Palm oil	34,688,841		

Exports of Foreign Fats and Oils, Quarter Ended Mar. 31, 1926

Kind	Pounds	Kind	Pounds
Beef and hog fats	15,575	Olive oil, edible	55,681
Fish oils	64,280	Palm & palm-kernel oil	978,850
Other animal oils, fats and greases	10,109	Peanut oil	14,067
Chinese nut oil	940,868	Soya-bean oil	182,600
Coconut oil	580,993	Other vegetable oils	36,954
		Vegetable wax	152,760

Exports of Domestic Fats and Oils, Quarter Ended Mar. 31, 1926

Kind	Pounds	Kind	Pounds
Oleo oil	22,479,093	Stearic acid	252,599
Neat's-foot oil	223,543	Other animal greases, oils, and fats	13,732,171
Other animal oils	230,986	Coconut oil	3,392,196
Fish oils	110,688	Cottonseed oil, crude	13,623,709
Oleo stock	2,466,530	Cottonseed oil, refined	4,681,858
Tallow	1,713,095	Linseed oil	481,457
Lard	206,285,197	Soya-bean oil	188,981
Lard, neutral	5,097,905	Corn oil	558,512
Lard compound, containing animal fats	4,247,758	Vegetable oil lard compound	2,371,093
Olco & lard stearin	1,817,841	Vegetable soap stock	2,942,945
Grease stearin	305,971	Other vegetable oils and fats	1,418,307
Oleic acid, or red oil	132,027	Glycerin	212,412

ABSTRACTS

Theory and practice of steam deodorisation of Saponifiable Oils. W. Brash, M.SC., A.R.C.S. (Lond.), A.I.C. **Journal of the Society of Chemical Industry,** March 26, 1926.

This is a carefully written article, going into the mathematics of a distillation of the volatile substances which give oils and fats their special flavors. Formulas are given showing the amount of steam required, pressures, temperatures, volumes, etc. Equations are worked out for the size of apparatus and almost every imaginable detail connected with the operation. The author states that the amount of odoriferous matter varies in different oils; it is undoubtedly low, and in the amount of one-tenth to five-tenths per cent. It gives the following summary of this paper which is too long to abstract:

(1) The pressure of the steam entering the deodoriser is approximately half the pressure of the steam supply.

(2) The temperature of the entering steam is given by the equation $T_1 = 0.87T_0$, and T_1 should be equal to T_2.

(3) The relative dimensions of the apparatus and the form of the outlet pipe may be calculated from the equations given in the paper.

(4) The concentration of the volatile matter at any moment is given by $X = ae - Kkt$.

(5) To shorten the time of deodorisation the most important factors are low pressure in the deodoriser, and high temperature of the oil.

Fat from spent (hydrogenation) catalyst. C. Stiepel. **Seifensieder-Ztg.,** 1925, **52,** 967-968; **Chem. Zentr.,** 1926, I., 1486. Considerable quantities (up to 1.8 per cent) of a brownish viscous fatty mass were obtained when catalyst residues were saponified to recover the fat in them. The product thus obtained consisted of oxidised fatty acids with a low iodine value and high saponification value. As no such compounds were obtained in the raw materials of the process, they must have been formed in the hydrogenation process. According to Normann, oxygen present in the hydrogen used could have this effect.

Composition of Olive Oil. K. Taufel and J. G. Sarria. **Anal. Fis. Quim.,** 1926, **24,** 25-40. The following data were obtained for a Spanish olive oil; $d^{15.5}$ 0-914, oleorefractometer reading at 25° 61-9 acid value, 0-62, saponif. value 192-6, iodine value (Winkler's method) 82-7, Reichert-Meissl value 0.13, Hehner value 95, m.p. of fatty acids 28.2°. It contained stearic acid 2.27 per cent, palmitic acid 7.55 per cent, oleic acid 83.94, linoleic acid 0.51 per cent, glycerol (calc.) 4.44 per cent, and unsaponifiable matter 0.79 per cent. Triolein and a-palmitodiolein were isolated.. Arachidic acid was absent.

Analysis of Soya-Bean Oil. H. Pfahler. **Chem. Unschau,** 1926, **33,** 65-70. A freshly prepared sample of soya-bean oil was found to have the following composition: linolenic acid 1.9 per cent, linoleic acid 29.2 per cent, isolinoleic acid (including a little isolinolenic acid) 24.3 per cent, oleic acid 30.8 per cent, stearic acid 7.0 per cent, palmitic acid 2.35 per cent, glyceryl residue (as C_3H_2) 3.88 per cent, and unsaponifiable matter 0.5 per cent. These numbers do not differ much from those of Baughman and Jamieson (B., 1923, 149 A), and yield a theoretical iodine value of 130, which is in close agreement with the value 132.4 obtained experimentally. To prepare linoleic acid the mixed fatty acids were brominated and the tetrabromide was separated and converted into the methyl ester of linoleic acid. The acid liberated from the ester had iodine value 172.4 (theoretical 181.4). This linoleic acid was again brominated; the resulting tetrabromide (m.p. 113°) corresponded to only 33 per cent of the original linoleic acid. It appears that in debrominating the bromide isomerisation of the acid takes place so that what is re-brominated is a mixture of isomeric linoleic acids leading to a solid bromide and to a liquid one.

Bromometric examination of fats. H. P. Kaufmann. **Z. Unters. Lebensm.,** 1926, **51,** 3-14; of B., 1925, 302. The activity of bromine in solution in various solvents is discussed and a 0.1 N-solution in absolute methyl alcohol saturated with wodium bromide is recommended for the determination of the iodine value of fats. Such a solution, probably due to the formation of sodium tribromide,

is very stable and loses only 1 per cent of its active bromine in 56 days. It is almost odourless and can be measured with a pipette without danger. The determination is simply and quickly performed with an accuracy comparing favorably with the Hanus method.

Rapid method of determining the iodine value with iodine and alcohol. W. Austen. **Pharm. Zentr.**, 1926, **67**, 209-210. In carrying out the method of Margosches, Hinner, and Friedmann (B., 1924, 639) it is of importance that 200 cc. of water be added to the fat solution, and that the minimum time of contact be 6 mon. Observing these precautions, lard, beef tallow, and linseed oil gave iodine values of 58, 35.5, and 172.8 against the Hubl values of 58, 36.4, and 173.4 respectively.

New value for milk-fat. J. Kuhlmann and J. Grossfeld. **Z. Unters. Lebensm.**, 1926, **51**, 31-42. The "Butyric acid value" is a modification of the Reichert-Meissl value and is a measure of the water-soluble volatile fatty acids of lower molecular weight than caprylic acid (coconut oil fatty acids). 5 g. of fat are saponified with 3 c.c. of potassium hydroxide solution (75 per cent) and 10 c.c. of glycerol, and the soap solution is diluted with 100 c.c. of water at 100°. The mixture is cooled to 20° and 50 c.c. of dilute sulphuric acid (2.5 per cent), 15 g. of anhydrous sodium sulphate, 10 c.c. of coconut oil soap solution (100 g. of coconut oil saponified with 100 g. of glycerol and 40 c.c. of 75 per cent potassium hydroxide solution and the solution diluted to 1 litre with water), and about 0.1 g. of kieselguhr, are added. After shaking for 10 min, the liquid is filtered and 125 c.c. of the filtrate are diluted with 50 c.c. of water and distilled with the usual precautions. The first 110 c.c. of the distillate, which contain about 96 per cent of the total butyric acid, are directly titrated with 0.1 N-alkali and from the result is subtracted that given by a blank without fat. The difference multiplied by 1.4, is the butyric acid value. The value for coconut-oil is 0.9 and for butter-fat 20, this latter being directly proportional to the Reichert-Meissl value. By the application of small corrections for coconut oil, the amount of milk-fat in admixture with other fats can be calculated from the butyric acid value and the saponification value to within an accuracy of 1 per cent.

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